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Combined Effects of Electro-Coagulation and Electro-Oxidation on Pesticide Wastewater, Using Iron Electrodes and Studying Current Variation and Electrode Gap

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Abstract: This study explores the combined use of Electrocoagulation (EC) and Electro oxidation (EO) to removal of persistent organic compounds from industrial wastewater, focusing on pesticide wastewater. The EC process efficiently removes colloidal and suspended particles within 45-60 minutes, achieving significant reductions in Chemical Oxygen Demand (COD), although some stable organic compounds remain. The EO process further breaks down these organic pollutants, reducing COD, Biochemical Oxygen Demand (BOD), Total Suspended Solids (TSS), pH, and iron concentrations, but requires more time, especially for colloidal particles. The hybrid EC-EO method first utilizes EC to eliminate charged species and then applies EO to degrade the remaining organic compounds, leading to the effective reduction of various pollutants within approximately one hour. The results indicate that iron electrodes were particularly effective in removing pollutants under different treatment conditions. The combined EC-EO treatment method demonstrates significant potential for the efficient treatment and separation of contaminants in industrial wastewater, offering a promising solution for improving water quality and reducing environmental pollution.
Keyword: Electro-coagulation, Electro-oxidation, BOD, COD, TOC.

Graphical Abstract

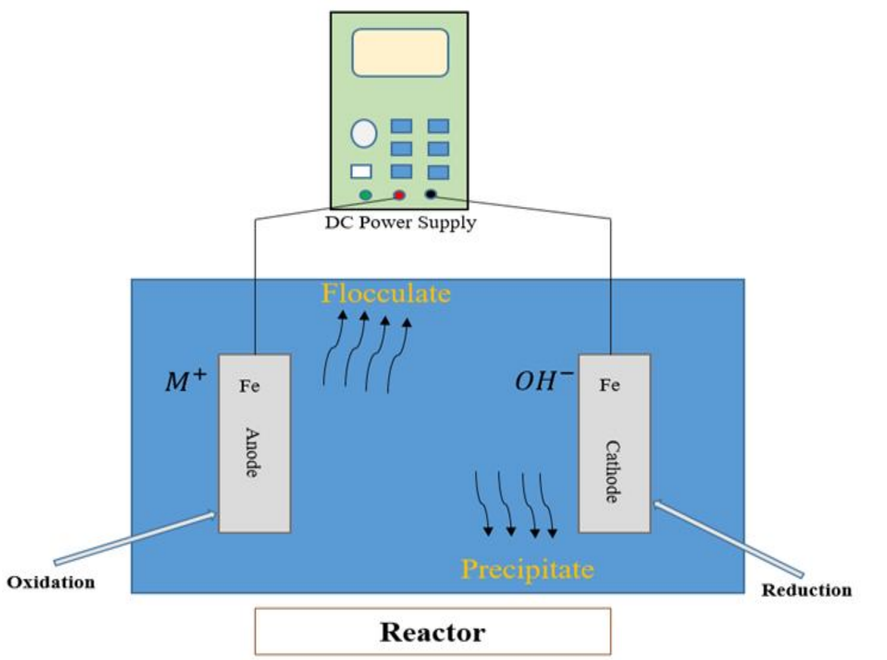

Fig.1

I. INTRODUCTION

Fresh water is essential for human survival. However, a World Health Organization report states that over 2.1 billion people do not have enough safe water daily, leading to 3.4 million deaths each year from water scarcity or contamination. This makes the freshwater crisis the fourth most serious threat to humanity. The report also highlights that 3 in 10 people lack access to safe water at home, and 6 in 10 lack proper sanitation. Many water-related diseases, which mostly affect children, are preventable with simple measures. For example, trachoma, a leading cause of blindness, occurs in areas without proper water and sanitation [1].

Industrial effluents have many pollutants, including bio-refractory organic compounds that are hard to treat. These compounds can be toxic and cause taste and odour issues in water. Biological treatment alone does not remove them, requiring extra physical or chemical treatments to improve water quality and biodegradability [2]. The treatment of industrial wastewater is challenging because it varies greatly in volume and composition, contains high levels of organic matter and salts, and includes hard-to-treat compounds. Biological treatment faces issues like poor separation of bio-solids, excessive sludge, and low efficiency. Treatment facilities need to enhance their performance and meet stricter quality standards [3].

EC is a process where coagulants are created by dissolving aluminium or iron ions from metal electrodes using electricity. This happens at the anode, and hydrogen gas is produced at the cathode. The metal ions combine to form flocculates that trap contaminants, while the hydrogen gas helps float these particles. EC is also known as electro-flocculation. It has been effectively used to remove phenolic compounds, decolorize dye solutions, clarify clay solutions, treat textile wastewater, and eliminate heavy metals. The benefits of EC include high efficiency in removing particles, a compact treatment setup, and low cost for full automation [2]. EC employs electrons as the main reagents, which makes it a clean method that does not need extra chemicals. In essence, an EC reactor consists of an electrolytic cell with one anode and one cathode, featuring conductive metal plates known as "sacrificial electrodes". The process produces destabilization agents like Aluminium and iron to neutralize electric charges and remove pollutants. EC leads to less sludge production, no chemical requirements, and simple operation, with successful applications in various wastewaters, including tannery and petroleum refinery wastewater [3].

In EO, pollutants are broken down through either direct or indirect oxidation. In the direct method, pollutants stick to the anode surface and are eliminated by a reaction involving electrons from the anode. In the indirect method, strong oxidants like hypochlorite, ozone, and hydrogen peroxide are generated through electrochemical processes. These created oxidants then react with the pollutants in the solution, effectively destroying them. The oxidants are produced on-site and used right away [4].

EO has been used to treat wastewater from various sources, including gelatine production [5], alcohol oxidation in water [6], and different industrial wastewaters such as distilleries and chemical industries [7, 8]. There is growing interest in electrochemical techniques to eliminate hard-to-treat organic compounds in industrial wastewater. In the indirect process, chlorine and hypochlorite generated at the anode are used to clean organic pollutants. Another way to break down contaminants is through hydrogen peroxide generated electrochemically. Direct anodic oxidation is also possible, which uses "active oxygen" generated on the anode without needing extra chemicals or oxygen, making the process simpler and not creating secondary pollutants.

The anode is crucial in this process, with common materials including glassy carbon, titanium, carbon fiber, and stainless steel. However, these materials often lack a good balance of activity and stability. Recently, boron-doped conductive diamond anodes have emerged as promising for treating organic waste due to their higher stability and efficiency, making them useful in various applications, including the complete mineralization of organic waste [9, 10]. The advantages of EC include high particulate removal efficiency, a compact treatment facility, relatively low cost, not requiring supplementary addition of chemicals, and reduces the volume of produced sludge and the possibility of complete automation [11]. Oxidation of iron in an electrolytic system creates iron hydroxide, Fe (OH) n, where n = 2 or 3. Two mechanisms have been proposed for producing Fe (OH) n; one is shown below with more details provided [12].

At Anode:

$$4Fe_{(s)} \rightarrow 4Fe^{2+}_{(aq)} + 8e^{-}$$

$$4Fe^{2+}_{(aq)} + 10H_2O_{(l)} + O_{2\,(g)} \rightarrow 4Fe(OH)_{3\,(s)} + 8H^{+}_{(aq)}$$

At Cathode:

$$8H^{+}_{(aq)} + 8e^{-} \rightarrow 4H_{2\,(g)}$$

Overall:

$$4Fe_{(s)} + 10H_2O_{(l)} + O_{2\,(g)} \rightarrow 4Fe(OH)_{3\,(s)} 4H_{2\,(g)}$$

The Fe (OH) $_{n\ (s)}$ formed remains in the aqueous stream as a gelatinous suspension; either can remove the pollutants from wastewater either by complexation or by electrostatic attraction, followed by coagulation [12]. In this study, we present the results of using EC and EO on industrial wastewater. The main goal is to compare how effective each method is at removing pollutants. After finding the best conditions, we combine them to achieve better pollutant removal. The study aims to treat industrial wastewater to eliminate COD, TOC, colour, and turbidity.

II. EXPERIMENTAL STUDIES

A. *Electro-coagulation Treatment*

A batch mono-polar electrochemical cell was use for EC, with a capacity of 0.350 litres. Iron served as both the anode and cathode electrodes, each measuring 0.12 m long and 0.11 m wide, resulting in an area of 0.0132 m². Wastewater was measure and placed in a reactor. A DC power supply provided current densities of 200 to 800 A/m² without additional electrolytes. Samples were filter and analysed for TOC, TSS, TDS, pH, COD, and colour at different electrode gaps and currents. Faraday's Law was use to calculate the maximum amount of iron produced in the electrochemical process. The calculation considered a current intensity of I = 0.5–5 A and t = 60 min, along with the Faraday Constant (F = 96,500 C/mole) and the charge on the cation (z = 2^+).

$$n = \frac{It}{ZF} \qquad \text{........ (1)}$$

The iron concentration in the solution was calculate by using

$$Fe = \frac{n}{V} \qquad \text{.......... (2)}$$

Where n is the amount of iron in moles, and V is the volume of the cell in litres (0.350L).

B. *Electro-oxidation Treatment*

The EC process was done in batch cells using iron as both the anode and cathode. The electrodes were rectangular plates with an area of 0.0132 m2. Treated wastewater was stored in a 0.250 L glass tank, and the process was powered by a DC source at 0.5–5 amps, creating a current density of 200–800 A/m2 within a pH range of 5 to 9. However, the pH was not properly maintained during treatment, and no extra electrolytes were added during electrolysis.

C. *Waste-Water Samples*

Wastewater samples were collected at a treatment plant in an industrial zone of Meerut District, Uttar Pradesh, India. This plant receives industrial waste from various factories, including those in chemicals, metal finishing, textiles, food processing, pharmaceuticals, automobiles, and leather. The plant treats all the industrial effluents together. It features biologically active sludge reactors, primary clarifiers, sand separators, oil and grease separators, secondary clarifiers, and a chlorine disinfection unit. However, this treatment only removes about 40% - 50% of the COD, BOD, TSS, TDS, and pH levels, indicating that extra treatment steps are necessary to enhance wastewater quality. After the primary treatment, wastewater samples were collect in plastic containers and stored at 4^0C for analysis and electrochemical treatments.

1) *Physicochemical characteristics of wastewater*

The wastewater samples were collect after primary treatment to create an electrochemical test with DC source. The samples contained a high range of pollutants and organic compounds as shown in **Table 1**. The COD is twice the TOC, indicating a 2:1 composition of the wastewater, likely due to high levels of pollutants like total dissolved solids, BOD, Iron, acidity and alkalinity, complicating the oxidation of organic matter [3].

Table 1. Physicochemical Characteristics of Raw Wastewater

S.No.	Parameter	Results	Unit
1	pH	8.90	…..
2	BOD	167.5	mg/l
3	COD	4200	mg/l
4	TOC	102.4	mg/l
5	TDS	5627	mg/l
6	EC	131.2	ms/cm
7	Fe	1.70	mg/l
8	DO	<4.0	mg/l

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D. Methods of Analysis

The treatments were evaluated by analysing various factors such as COD (Total Oxygen Demand), TOC (Total Organic Carbon), Colour, Turbidity, pH, Alkalinity, Acidity, Hardness, Sulphates, Ammonia, Nitrogen, Chlorides, Nitrates, and Total Phosphorus, following Standard Methods and Procedures. Each treatment also included testing BOD (Biochemical Oxygen Demand), TSS (Total Suspended Solid), TDS (Total Dissolved Solids), Dissolved Iron Concentration, and Electrical Conductivity.

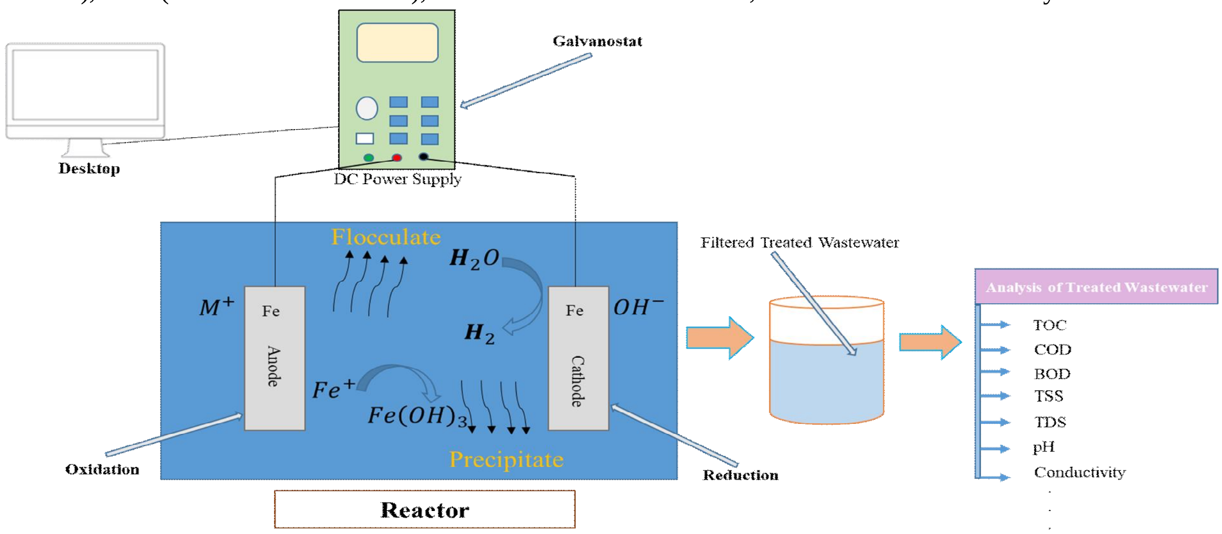


Fig. 2 Process Diagram

E. Simulation of Owon SPE32102 Galvanostat

1) SPE Series Single Channel Digital DC Power Supply

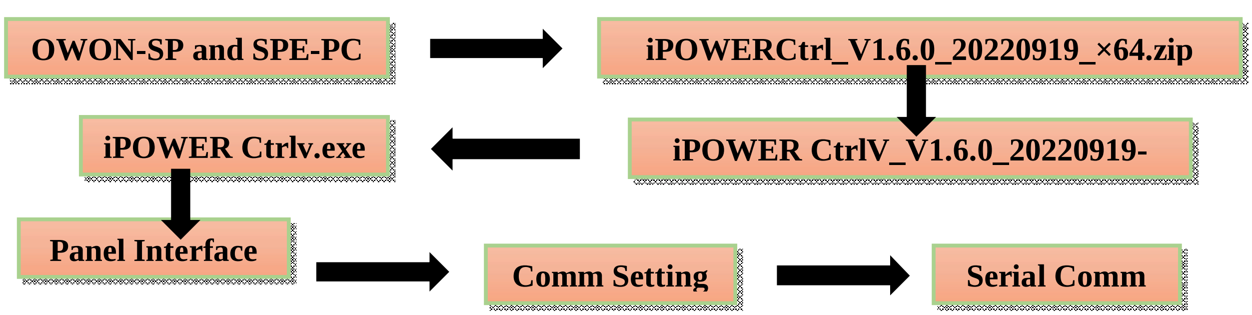
The Owon SPE3102 is a compact, single-channel DC benchtop power supply featuring over-voltage protection (OVP) and over-current protection (OCP). It offers high resolution at 10mV/1mA and has a 2.8" LCD that shows set, limit, and momentary parameters. Users can monitor real-time voltage and current using graphs. The device can store and edit up to four output waveforms. It connects to a PC via USB and communicates through SCPI. The power supply has an ultra-thin body, is portable, adjustable up to 30V and 10A, and includes various protection and cooling features, making it suitable for beginners.

2) How to Operate OWON SPE 3102

To operate the OWON SPE 3102, follow these steps to check the basic current functions with a short across the power supply's output:

- Connect a short across the (+) and (-) output terminals using an insulated test lead appropriate for maximum current.
- Set the output voltage to the maximum rating for the channel.
- Turn on the channel output, ensuring it is in Constant Current output mode.
- Set different current values on the channel and check if the displayed actual current is close to the set value, and the actual voltage is nearly zero.
- Verify if the output current adjusts from zero to the maximum rating, a beep should sound when limits are reach.
- Finally, turn off the channel output and remove the short circuit.

3) Procedures to Run Programmable OWON SPE3102 (Galvanostat)



F. Experimental Procedure

Turn on the power supply of the Galvanostat and Desktop. Measure 350 ml of the effluent in a beaker and pour it into the glass reactor with dimensions (11 X 2. 5 X 14) cm^3. Start the process, connect the Galvanostat to the reactor, and operate for 1 hour. The distance is constant at 1mm, and the current varies at 0.5 amp, 1 amp, 1.5 amp, 4 amp, 4.5 amp, and 5 amp. After another hour at 5 amp, change the electrode gap to 1mm, 3mm, and 5mm. Use SS (iron) electrodes for the anode and cathode fixed at 1mm. After 1 hour, stop the operation, discharge the effluent for filtration, use filter paper, and store the filtered water in a sample bottle. Weigh the wet filter paper with the solid residue.

Fig.3 Galvanostat Setup

Fig.3 Upper and Side View of Glass Reactor and SS Electrode during Experiments

III. RESULT AND DISCUSSIONS

A. Effects of Currents

The current affects the amount of coagulant needed and the speed and size of bubble production in EC treatment for pollutant removal. The study looked at different currents (0.5-5 amp) on chemical oxygen demand (COD), biological oxygen demand (BOD), and total organic carbon (TOC) removal as shown in Figure 4. Higher currents improved treatment efficiency by generating more small hydrogen bubbles, aiding sludge separation [3, 14].

Samples went through EC to reduce suspended solids and COD. Ions like Fe2+ formed effective coagulants by destabilizing suspended solids. COD dropped significantly from 4200 mg L−1 to 200 mg L−1. Most COD-related suspended solids were removed quickly, with higher currents speeding up coagulation. Total nitrogen also decreased, with maximum pollutant reduction occurring at 3 amp, preparing the sample for further studies [3, 14].

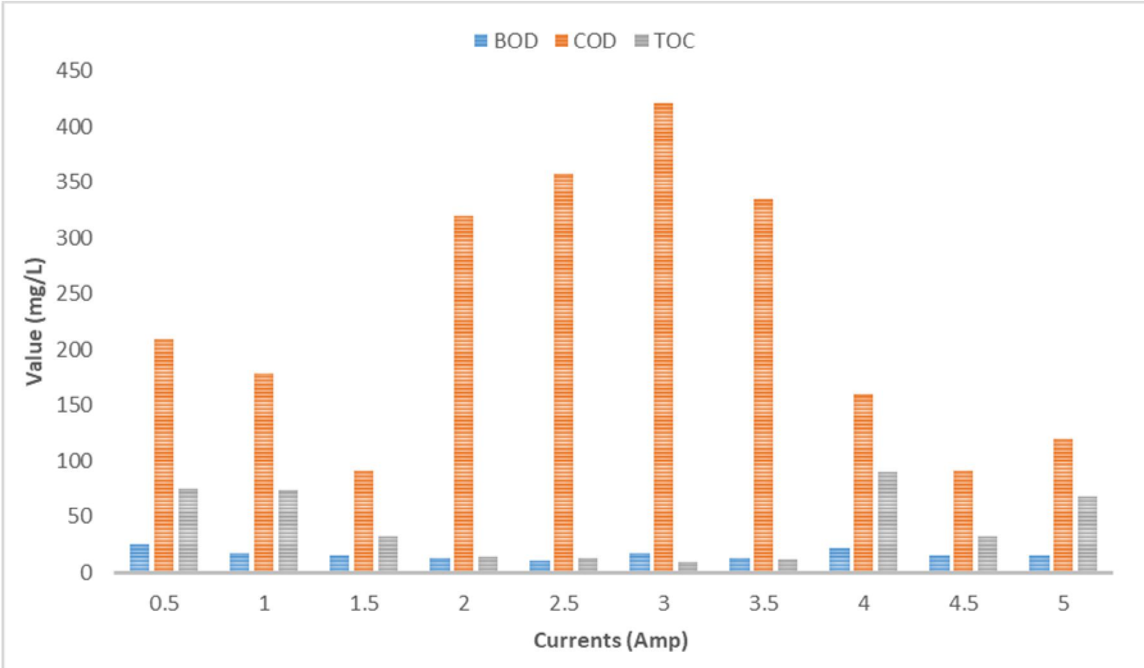


Fig. 4 Effects of currents on COD, BOD, and TOC

B. *Effects of Electrode Gap*

The electrode gap affects the amount of coagulant needed and the speed and size of bubble production in EC and electro-oxidation treatment for pollutant removal. The study looked at different electrode gaps (1-5 mm) on chemical oxygen demand (COD), biological oxygen demand (BOD), total dissolved solids (TDS), total organic carbon (TOC), dissolved iron (Fe), electrical conductivity and pH removal as shown in figure 5. Higher electrode gap improved treatment efficiency by generating more small hydrogen bubbles, aiding sludge separation. Samples went through EC to reduce suspended solids and COD. Ions like Fe2+ formed effective coagulants by destabilizing suspended solids. Most COD-related suspended solids were remove quickly, with higher currents speeding up coagulation. Total nitrogen also decreased, with maximum pollutant reduction occurring at 5 mm, preparing the sample for further studies.

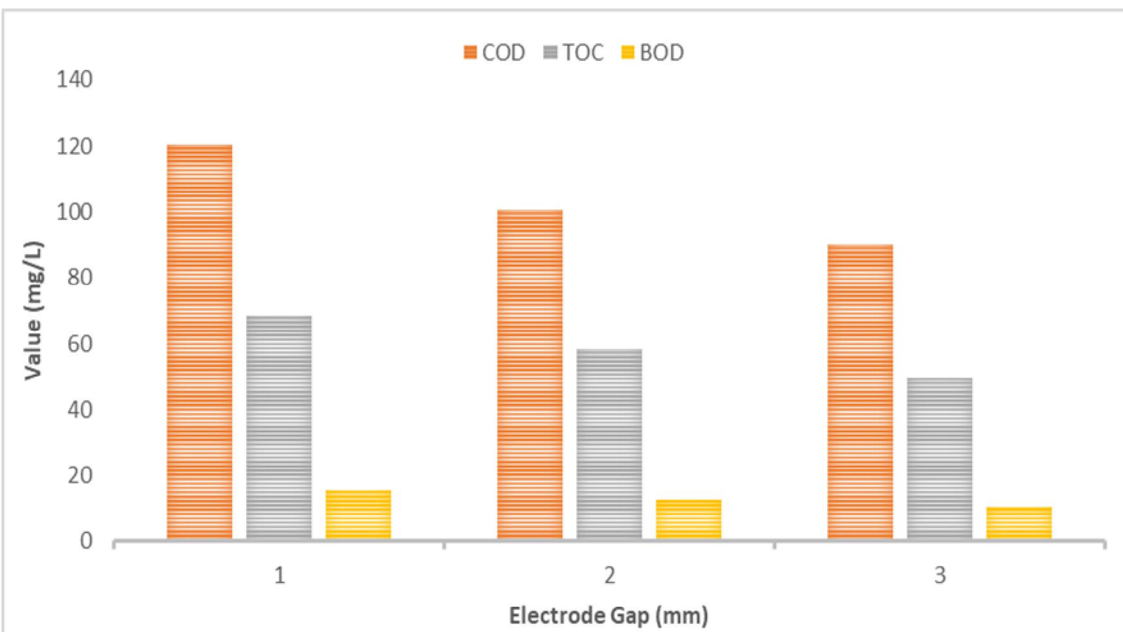


Fig. 5 Effects of electrode gap on COD, BOD, and TOC

C. Effects of EC and EO on various parameters of water

The treated water from the EC process underwent EO treatment by applying a current between 0.5-5 A and an electrode gap of 1-5 mm. Tests showed that COD reduction efficiency increased with current, helping to select a reasonable current for operation. This approach achieved COD discharge values that meet legal limits quickly and without high operational voltage. At 60 minutes of EO treatment at pH 6 to 8, COD dropped below the acceptable level, with the best pH for treatment being between 6.5 and 7.5. Iron electrodes were use after EC to treat pesticide effluents, showing positive results and indicating that combining EC and EO is effective for wastewater treatment [15].

IV. CONCLUSIONS

The Electrocoagulation (EC) and Electro oxidation (EO) technique proves to be highly effective in treating and separating contaminants present in industrial wastewater. This technology demonstrates significant potential for treatment, with electrochemically generated iron concentrations varying from 2.12 to 1.21 mg/l. The following conclusions can drawn from the findings.

Iron electrodes exhibited superior performance in pollutant removal from pesticide wastewater at a current of 3 Amperes. After 60 minutes of treatment, with pH adjustments and a consistent electrode gap of 1 mm, the removal efficiencies were as follows: BOD (89.7%), COD (89.96%), TOC (90.03%), Electrical Conductivity (71.41%), pH (86.29%), and Iron concentration increased by 17.64%. Conversely, when utilizing an electrode gap of 5 mm and maintaining a constant current of approximately 5 Amperes for 60 minutes, the removal efficiencies improved further: BOD (93.77%), COD (97.85%), TOC (51.56%), Electrical Conductivity (55.07%), pH (83.70%), and Iron concentration reduced by 45.88%.

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